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## **Consolidated Canadian Results to the HEU Round Robin Exercise**

*Performed under the auspices of the Nuclear Smuggling  
International Technical Working Group (ITWG)*

Carey L. Larsson and Dean S. Haslip

**Defence R&D Canada – Ottawa**

TECHNICAL MEMORANDUM

DRDC Ottawa TM 2004-192

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Canada

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## Abstract

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In the framework of combating the illicit trafficking of nuclear materials and other radioactive substances, the ITWG organised an international inter-laboratory round robin dealing with a thwarted illicit trafficking incident of a high-grade sample of highly enriched uranium (HEU). DRDC Ottawa coordinated Canada's response to the exercise, which included the participation of seven organisations, including this lab. The exercise involved a thorough forensic examination and analysis by the Royal Canadian Mounted Police, nuclear characterisation of the material by six laboratories, and interpretation of the nuclear data for attribution of the materials origin by the Canadian Nuclear Safety Commission. Overall, the analysis was successful in identifying the material and providing a reasonable interpretation of the data. This report outlines Canada's response, the lessons learned, and the way ahead in terms of combating illicit trafficking and nuclear smuggling in Canada. A comparison of Canada's results to those of the other participating countries is also presented.

## Résumé

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Dans le cadre de lutter contre le trafic illicite des matériaux nucléaires et d'autres substances radioactives, une évaluation comparatif inter-laboratoires international a été organisé par l'ITWG basé sur un incident de trafic illicite d'uranium hautement enrichi (UHE) intercepter par une agence judiciaire. RDDC Ottawa a coordonné l'intervention canadienne à l'exercice, qui a inclus la participation de sept organismes, y compris ce laboratoire. La Gendarmerie Royale du Canada a exécuté une analyse judiciaire, suivi par la caractérisation nucléaire du matériel par les autres six laboratoires, et l'interprétation des données pour l'attribution d'origine de matériaux par la Commission canadienne de sûreté nucléaire (CCSN). En tout, l'identification du matériel et l'interprétation des données a été une réussite. Ce document résume l'intervention du Canada, les leçons apprises, et le chemin à suivre pour combattre le trafic illicite et la contrebande nucléaire au Canada. Une comparaison des résultats canadienne à ceux des autres pays est aussi présentée.



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## Executive summary

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*Introduction:* The Nuclear Smuggling International Technical Working Group (ITWG) organized an exercise using a highly enriched uranium (HEU) sample. Canada began its portion of the exercise in April 2004 with the participation of seven laboratories. The round robin tested both forensic and nuclear analysis capabilities of the groups involved, as well as the interconnections between the labs. Participation in this exercise was intended as a collaborative learning experience for the law enforcement and scientific communities, and not as a performance evaluation of the individual laboratory participants.

*Results:* In comparison to the other participating countries, the Canadian response was on par. All of the countries performed well in some areas of the response and not so well in other areas. Overall, Canada's response was successful in identifying the material and providing a reasonable interpretation of the data. As this was the first such exercise of its kind in Canada, several shortcomings became apparent, including:

- Transport delays and Chain of Custody concerns
- Reporting delays and deficiencies from several of the laboratories
- Requirement for protocols describing the handling of radioactively contaminated forensic evidence
- Results discrepancies between laboratories

*Significance and Future Plans:* As the first exercise of a nuclear forensic and illicit trafficking incident in Canada, there were several "lessons learned". Addressing these concerns and performing further inter-comparisons will certainly prepare the appropriate individuals and organizations for a real incident in Canada.

Larsson CL and Haslip DS. 2004. Consolidated Canadian Results to the HEU Round Robin Exercise - Performed under the auspices of the Nuclear Smuggling International Technical Working Group (ITWG). DRDC Ottawa TM 2004-192. Defence R&D Canada – Ottawa.

## Sommaire

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Introduction : Le Group de travail Technique International de contrebande nucléaire a organisé un exercice avec un échantillon d'uranium hautement enrichi (UHE). Le Canada a commencé sa portion de l'exercice en avril 2004 avec la participation de sept laboratoires différents. Le test comparatif a examiné la capacité des laboratoires pour faire une analyse judiciaire et une caractérisation nucléaire du matériel, et aussi les intercommunications entre les laboratoires. La participation à cet exercice a été prévue comme une expérience d'apprentissage pour les services policiers et les communautés scientifiques, et non comme une évaluation de la performance des laboratoires participants.

Résultats : En comparaison avec les autres pays participants, l'intervention canadienne était égale aux autres pays. Tous les pays ont bien exécuté dans quelques secteurs de l'intervention et n'ont pas aussi bien performé dans d'autres secteurs. Généralement, l'intervention du Canada a réussi à identifier le matériel et à fournir une interprétation des données. Puisque c'était le premier exercice de la sorte au Canada, plusieurs faiblesses sont devenues évidentes, incluant :

- Des délais dans le transport et des soucis avec la continuité de possession
- Des délais dans la communication des rapports et des faiblesses de plusieurs laboratoires
- L'exigence des protocoles décrivant la manipulation de l'évidence judiciaire qui a été contaminée radiologiquement.
- Les différences des résultats entre différents laboratoires

Importance : Étant le premier exercice d'un incident de trafic illicite de matériaux nucléaires au Canada, il y a eu plusieurs leçons tirées. L'adressage des soucis et l'exécution d'autres comparaisons corrélative prépareront les individus et les organismes appropriés pour un incident réel au Canada.

Larsson CL et Haslip DS. 2004. Consolidated Canadian Results to the HEU Round Robin Exercise - Performed under the auspices of the Nuclear Smuggling International Technical Working Group (ITWG). DRDC Ottawa TM 2004-192. R & D pour la défense Canada – Ottawa.

## Table of contents

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Abstract.....	i
Executive summary .....	iii
Sommaire.....	iv
Table of contents .....	v
List of figures .....	vi
1. Background.....	1
2. Objectives of the Round Robin .....	2
3. Canada's HEU Round Robin Response .....	5
3.1 24-hour Response .....	5
3.2 One-week Results.....	9
3.3 Two-month Results .....	11
4. Comparison to ITWG results.....	13
5. Discussion .....	17
6. Recommendations and Conclusions.....	20
7. References .....	22
Annex A - Scenario .....	23
Annex B – ITWG Expectations.....	27



## List of figures

---

Figure 1. Forensic evidence for HEU Round-Robin Exercise. Evidence included (from lower left clockwise) finger prints on vials, a plastic shopping bag with vials and a coaster with a map attached, pollen seeds in a vial, the HEU oxide sample, and a beer-stained coaster with directions on the back.....	2
Figure 2. Documentation of package exterior. (a) Radiation measurements and information documentation. (b) Close-up of package seal. ....	6
Figure 3. Gold-coloured canister containing evidence material, shown (a) unopened and (b) opened with contents removed. ....	6
Figure 4. Explosives detection using foXray machine. (a) Radiography unit and (b) Visual display. ....	7
Figure 5. Scanning electron microscopy (SEM) image of HEU sample (bar shows 5 $\mu$ m). ....	9
Figure 6. Fingerprints of suspect taken on the scene. ....	24
Figure 7. Pictures taken on the scene. (a) Confiscated material. (b) Inside view. ....	25

## List of tables

---

Table 1. Canada's HEU round robin participants.....	3
Table 2. Worst-case 50 year Committed Effective Doses.....	8
Table 3. Elemental and isotopic composition of HEU sample.....	10
Table 4. Composition of HEU sample.....	12
Table 5. HEU isotopic analysis results comparison (in atom %) .....	15
Table 6. Attribution of HEU material history - Nuclear processes .....	15
Table 7. Technique and method prioritization for HEU round robin exercise .....	16
Table 8. Analyses promised, performed and used in the exercise reports.....	19

# 1. Background

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In the framework of combating the illicit trafficking of nuclear materials and other radioactive substances, a Nuclear Smuggling International Technical Working Group (ITWG) was created in 1996 under the auspices of the P-8 Non-Proliferation Experts Group (NPEG) [1]. Amongst the objectives of this international technical and scientific group, the following elements were recognized as a high priority for development to assist competent bodies (police, customs, etc.) in their investigations:

- Development of protocols for collection and preservation of evidence that meets the requirements of specialized measurements; in addition, also develop protocols for laboratory investigation.
- Evaluations and recommendations regarding technical equipment for initial hazard evaluation and on-site assessment of nuclear material composition.
- Prioritize techniques and methods for forensic analyses of nuclear and non-nuclear materials associated with illicit nuclear materials trafficking in order to answer questions regarding source attribution, route attribution, and intended use of the nuclear materials.
- Development of forensic databanks to assist in the interpretation of analytical results.
- Facilitate technical assistance to countries (including non G-8 countries) in response to specific requests.
- Formulate and execute inter-laboratory exercises to evaluate and improve the effectiveness of forensic techniques and methods.

In relation to the last of these priorities, two inter-laboratory exercises have been held. The first was launched in 1999 with the participation of six countries and dealt with isotopic and elemental analysis of plutonium. Canada did not take part in this round robin. The second exercise, using a highly enriched uranium (HEU) sample, began in 2000 with the initial participation of nine countries. Canada was recently invited by the chair of the ITWG to retroactively participate in the exercise, becoming the tenth country involved.

In this report, Section 2 describes both the ITWG and Canada's objectives of the round robin, Section 3 describes Canada's overall response and provides a summary of the key findings and results from the exercise, Section 4 compares Canada's results with the nine other participating countries, Section 5 discusses shortcomings related to Canada's response, and Section 6 provides a summary of conclusions and recommendations pertaining to the exercise. The scenario for the exercise is provided in Annex A.



## 2. Objectives of the Round Robin

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The ITWG goal for nuclear forensics is to develop a widely understood and accepted approach to support illicit-trafficking investigations [2]. As such, the objectives of the two international inter-laboratory exercises organized and executed by the ITWG were to evaluate and improve the effectiveness of current forensic techniques and methods. These exercises were organized to have the broadest possible international contribution in order to compare nuclear forensic techniques from a wide variation in expertise.

Canada's main objective for participating in the Round Robin was to assess the various radiological/nuclear laboratories' inter-operability, evaluate attribution capabilities, and address any highlighted response shortcomings. This exercise tested the development of analytical techniques and interpretation of results generated at the individual laboratories. Participation in this exercise was intended as a collaborative learning experience for the law enforcement and scientific communities, and not as a performance evaluation of the individual laboratory participants.

The material used for this round robin exercise was a highly enriched uranium oxide powder, which was provided by the Czech Nuclear Research Institute in Rež. The history of the material was undeclared, but it was reported to be similar to materials seized in Prague in 1994. Non-nuclear forensic evidence was added to the sample and incorporated in the scenario in recognition of the importance of such "classical" forensic evidence in a real investigation. These items included fingerprints, pollen seeds, a plastic shopping bag containing the evidence, and a handwritten map on a beer coaster (see Figure 1). Canadian participants were told only that the sample consisted of HEU. The scenario is outlined in Annex A.



**Figure 1.** Forensic evidence for HEU Round-Robin Exercise. Evidence included (from lower left clockwise) finger prints on vials, a plastic shopping bag with vials and a coaster with a map attached, pollen seeds in a vial, the HEU oxide sample, and a beer-stained coaster with directions on the back.

Members of seven nuclear and non-nuclear laboratories participated in the analysis of the HEU, contributing a large variety of response capabilities. These laboratories are listed in Table 1, below. An eighth laboratory, the Laboratory and Scientific Services Division of the Canadian Border Services Agency (CBSA), also offered to participate in the non-nuclear analysis. However, delays in analysis at the RCMP lab prevented CBSA's participation. All of the labs were provided with the scenario, but no other information regarding the sample was given. This was done so as to treat the exercise as an actual nuclear smuggling analysis with the primary objective being to determine the nature and origin of the material in order to achieve a credible attribution capability.

Reporting of exercise findings to the ITWG was mandated at 24 hours, one week and two months following the exercise commencement. Special report forms provided included specific questions to be addressed for each period. The 24-hour report concentrated on details concerning the safety of responders and law enforcement personnel, public health and safety, and determination of criminal activity or immediate threat. An assessment of the degree of radiological threat of the material, including physical characterization and radiological attributes was also requested. The one-week report was focused on issues associated with the collection of evidence and preliminary forensic analyses to develop leads for the investigators. This report contained more detailed information on the physical characterization and isotopic, elemental, and chemical analysis of the sample to guide the investigation. The final report (2 months) required a comprehensive forensic analysis and attribution of the materials to provide new leads for investigation and evidence for prosecution.

**Table 1.** *Canada's HEU round robin participants*

LABORATORY	ANALYSIS TYPE	LIST OF ANALYSES
Defence R&D Canada – Ottawa (DRDC Ottawa)	Nuclear	<ul style="list-style-type: none"> <li>• Sample receipt and distribution</li> <li>• Radiological analysis</li> <li>• U isotopics (gamma spec)</li> </ul>
Royal Canadian Mounted Police (RCMP)	Non-nuclear	<ul style="list-style-type: none"> <li>• Forensics evidence processing (fingerprints, DNA, trace materials)</li> </ul>
Canadian Nuclear Safety Commission (CNSC)	Nuclear	<ul style="list-style-type: none"> <li>• U isotopics (gamma spec)</li> <li>• Interpretation</li> </ul>
Health Canada – Radiation Protection Bureau (HC-RPB)	Nuclear	<ul style="list-style-type: none"> <li>• U isotopics (gamma spec, ICP MS)</li> <li>• Trace elements</li> <li>• Particle composition and morphology</li> </ul>
Royal Military College SLOWPOKE Facility (RMC)	Nuclear	<ul style="list-style-type: none"> <li>• Neutron activation analysis</li> </ul>
University of Alberta SLOWPOKE Facility (UofA)	Nuclear	<ul style="list-style-type: none"> <li>• Neutron activation analysis</li> <li>• U isotopics (gamma spec, ICP MS)</li> </ul>
Defence R&D Canada – Atlantic Esquimalt Division (DRDC Atlantic)	Nuclear	<ul style="list-style-type: none"> <li>• U isotopics (gamma spec, ICPMS)</li> <li>• Trace elements</li> <li>• Particle composition and morphology</li> </ul>

This schedule of reporting was designed in light of experience with real cases. In an actual illicit trafficking incident, the first report would be necessary to provide information rapidly in order to enable the responsible constabularies to decide how to address the case and decide whether suspected smugglers should be detained. Timely transmission of the second report to the police and legal authorities, even at the political level, is of great importance and, in practice, should occur within 1 week of receipt of seized material. The third report would be necessary to provide investigators with as much forensic data as possible to support the investigation and to provide the crown with the evidence necessary to prosecute the crime.



### **3. Canada's HEU Round Robin Response**

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Prior to the commencement of the exercise, details concerning sample delivery and licencing had to be addressed. Since the sample was being shipped from Germany to Ottawa, applications for both an import permit (in Ottawa) and an export permit (in Germany) were necessary. This process took approximately two months to complete. Furthermore, several of the participating laboratories required modifications to their radiation licences in order to handle the HEU. With CNSC participation in this exercise, positively addressing these items was greatly simplified. The following sections outline the analysis of the HEU in the ITWG-specified reporting time periods.

#### **3.1 24-hour Response**

The official "Time Zero" for this exercise was set at 10h00 on April 27<sup>th</sup>, 2004, although the sample actually arrived at DRDC Ottawa during the previous week. Time shifting was done in order to accommodate some of the participating groups. A local transport carrier delivered the parcel to DRDC Ottawa stores (shipping/receiving) without further escort (i.e. law enforcement or CNSC accompaniment). Personnel in the Radiological Analysis and Defence Group were called in to perform a brief gamma survey of the package, which showed no radiation leaks. The package was then taken to a secure, locked radiation source vault located on the premises until the commencement of the exercise.

On the morning of the 27<sup>th</sup>, the RCMP were called in to DRDC Ottawa to perform a forensic examination of the package. The package remained in the vault until the RCMP arrived on scene. Personnel from the Explosives Disposal and Technology Section, and the Forensic Identification and Research Services performed a detailed inspection and forensic analysis of the package. The entire opening procedure was documented by both video and photography, and an outline of this procedure is given below.

Before opening the package, a sheet of Mylar was placed on the bench top to avoid cross-contamination of the evidence. All windows of room 33 in Building 5B of DRDC Ottawa were shut. The exterior of the package was photographed and information on the package was documented (see Figure 2). The outer package was a black metal canister of the type typically used for shipping radioactive materials. Knowing that the evidence had been packaged at another laboratory, no efforts were taken to look for explosives or other types of booby-traps when opening the exterior packaging. Personnel wore latex gloves at all times while handling the package. The handling and opening procedure was performed by two police officers – one (the 'dirty' officer) who dealt with the items as they were removed from the container and placed them into an evidence bag, held by the other (the 'clean' officer).



(a)



(b)

**Figure 2.** Documentation of package exterior. (a) Radiation measurements and information documentation. (b) Close-up of package seal.

Upon opening the container, a sheet of paper containing information on fingerprints of the apprehended suspect and a smaller gold-coloured sealed metal can were found. This can was removed from the packing canister and photographed (see Figure 3). Again it was decided that, since laboratory personnel must have played a role in packaging this can, there was no need for taking precautions with regards to explosives or booby-traps. The can was then opened with a can opener and the lid removed. An Exploranium MiniSpec and a TBM-3S alpha/beta probe were used to take a reading at the top of the opened can. While background counts in the room were negligible, the alpha/beta probe measured approximately 300 cps at the can opening. The MiniSpec reading was “not identified”, with a suggested identification given as Uranium-235, Technetium-99 (mistaking a U-235 peak), and Potassium-40 (naturally occurring). The can was found to contain a white plastic grocery bag (that contained a small piece of cardboard) sealed in a double clear plastic bag; and two vials, packaged individually



(a)



(b)

**Figure 3.** Gold-coloured canister containing evidence material, shown (a) unopened and (b) opened with contents removed.



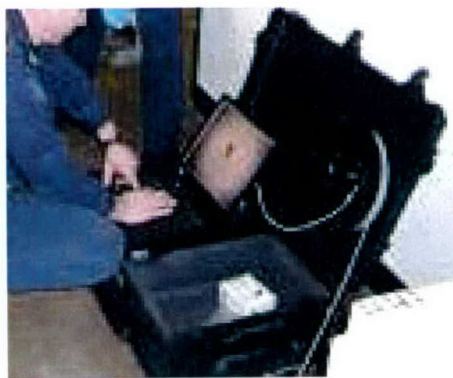
in both an inner Ziploc bag and two sealed clear plastic bags. No activity was measured from the sealed white plastic bag or one of the sealed vials. The second vial was radioactive and a MiniSpec reading of this item identified the material as Uranium-235, Gallium-67 and Technetium-99 (the two latter of which have peaks near U-235). These mis-identified peaks indicate that the MiniSpec device is far from foolproof and that operator knowledge is important for interpretation of measurements taken with the device.

It was assumed that neither of the vials had been opened during the initial lab analysis, and therefore both vials were radiographed using the RCMP's Vidisco (Petach Tikva, Israel) FoXrayII portable X-ray inspection system to assess the presence of explosives or other booby-traps. Each vial was radiographed separately. The non-radioactive vial appeared empty and no sign of booby-trap was seen. In contrast, the second vial, which was the vial emitting radiation, appeared to be full of some material. However, no evidence of booby-traps were seen (see Figure 4).

In order to rule out the presence of loose contamination within the sealed plastic bags before taking the evidence to the RCMP laboratory for further forensic analysis, the two vials were separately placed into a small glove box for opening. The non-active vial was assessed first, using both a Technical Associates (Canoga Park, CA) TBM-3S alpha/beta probe and a Graseby Dynamics (Watford, UK) Enhanced Chemical Agent Monitor (ECAM). No contamination was measured with either the ECAM or the alpha/beta probe, so the vial was placed into an evidence bag and taken by the RCMP for further analysis. The active vial was then placed into the glove box and assessed for contamination. The Ziploc bag containing the vial appeared worn, but otherwise nothing unusual was seen. A swipe of the ridged edge of the cap of the vial was taken and analysis of the swipe indicated significant levels of radiological contamination on the vial. Assessment of the Ziploc bag with the alpha/beta probe indicated radiological contamination also, and thus the RCMP did not remove either of these items from DRDC Ottawa for further analysis.



(a)



(b)

**Figure 4.** Explosives detection using foXray machine. (a) Radiography unit and (b) Visual display.



With the inspection of the material complete, the RCMP removed all non-nuclear forensic evidence for further analysis at their laboratory. The active vial was double bagged and DRDC Ottawa personnel performed gamma spectrometry analysis on the unopened vial with an 80% efficient ORTEC (Oak Ridge, TN) GMX-80 hyperpure germanium detector system for 90 minutes. These results indicated that the material inside the vial was highly enriched uranium with at least 80% Uranium-235 enrichment, by mass. However, it was expected that the brass container was shielding the low energy gamma rays significantly. This effect would cause an underestimation of the level of enrichment due to the low energy gammas used to assess U-235 and U-234 content in the sample.

The last task to be carried out in the first 24 hours of the exercise was sample division for each of the participating laboratories. Division of the sample was performed in a sealed glove box due to the powdered nature of the sample. Six aliquots were prepared, as well as a microscope slide and an LSC sample. Sample division also allowed for a visual inspection of the HEU, indicating that the material was a dark-coloured powder with a non-homogenous particle size distribution. The aliquots were then packaged for transportation to the other labs.

All of this information was portrayed in the 24-hour report, along with information regarding the threat posed by the material. The powdered nature of the sample along with the preliminary gamma spectroscopy results indicating at least 80% enriched uranium allowed for the assessment that this material constituted a threat, especially in large (kilogram) quantities, due to the fact that it could be used to construct an improvised nuclear device. In terms of the material seized, the powdered nature of the material combined with the fact that it emits both alpha and beta radiation suggest that this material poses a serious inhalation and ingestion hazard if the vial were to be opened. Based on this, 50-year committed effective doses were calculated for the worst-case scenarios for both inhalation and ingestion, shown in Table 2 below.

In regards to the expectations of analysis in the first 24 hours, Canada's response was more than adequate. While the time was mainly spent performing the forensic investigation, initial radiation measurements were made providing insight into the threat posed by the material. Contamination on the exterior of the vial was identified and proper precautions were taken.

**Table 2. Worst-case 50 year Committed Effective Doses**

ISOTOPE	METHOD	EFFECTIVE DOSE (mSv)
<sup>234</sup> U	Inhalation	530
<sup>234</sup> U	Ingestion	2.764
<sup>235</sup> U	Inhalation	559
<sup>235</sup> U	Ingestion	3.055
<sup>238</sup> U	Inhalation	17.8
<sup>238</sup> U	Ingestion	0.099

### 3.2 One-week Results

The second reporting phase for this exercise focussed on a comprehensive nuclear analysis of the sample. Arrival of the sample aliquots at each of the participating nuclear laboratories occurred at staggered times. The two local labs, Health Canada and CNSC, both received their samples within a couple of hours, since a CNSC inspector personally delivered them. The three other aliquots were shipped with a courier service (as overnight delivery) and took several days to arrive at the labs (DRDC Atlantic, U of A, & RMC). In a real nuclear smuggling scenario, all of the samples would need to be sent under higher security due to chain of custody issues. This was particularly apparent in the delivery of RMC's sample, where the transport driver handed off the sample without asking for identification or verification of any kind. The proper delivery channel would likely be either through a CNSC inspector or RCMP officer.

In follow-up to the forensic analysis, fingerprint analysis of the contaminated plastic Ziploc bag that contained the radioactive vial was performed during the first week. The analysis involved placing the bag in a sealed plastic fume box. A cyanoacrylate smoke was injected into the box, which has the property of polymerizing to fingerprints, making them more visible. The box was then opened, the fumes allowed to disperse and both the control and the evidence bags were analysed. The radioactive bag had no visible fingerprints on it. Forensic analysis of the other evidence was not completed during the first week.

Visual characterization of the sample was performed first via optical microscopy, then using scanning electron microscopy (SEM) by Health Canada and DRDC Atlantic. SEM analysis provided the most detailed information, indicating that the grey-dark green coloured material consisted of agglomerates of microcrystalline material 1-3 microns in size. Further analysis (via SEM) revealed that the small particles a few microns in size originally identified were agglomerates of still smaller particles no larger than 10 to 20 nanometres (see Figure 5).



**Figure 5.** Scanning electron microscopy (SEM) image of HEU sample (bar shows 5 $\mu$ m).



Elemental and isotopic characterisation of the sample was performing with high-resolution gamma spectrometry by DRDC Ottawa, CNSC, and Health Canada, and also using inductively coupled plasma mass spectrometry (ICPMS) at Health Canada during the first week of the exercise. Determination of total uranium percentage in the sample was also performed by kinetic phosphorescence by Health Canada. Results for each procedure and each laboratory are summarized in Table 3 below. Inter-laboratory agreement was quite good for the uranium isotopic ratios with both methods. However, total uranium percentage measured via gamma spectrometry by Health Canada was significantly higher than that for both other methods. Furthermore, CNSC only analysed U-235 content.

Several interesting findings were reported with the High-Purity Germanium (HPGe) and ICPMS results.

1. Quantities of Pb-212, Bi-212 and Tl-208 were identified, all of which originate from Th-228 since the half-life of all daughters between Th-228 and Pb-212 are short lived. This Th-228 must be a product of U-232 or Th-232. However, Ac-228 was not detected in the sample, indicating that the Th-228 was not a decay product of Th-232 (since Th-232 decays to Th-228 via Ac-228).
2. ICP-MS analysis identified peaks at mass numbers 232 and 236; half-life considerations suggest that the A=232 peak derives either from U-232 or Th-232 (Pa-232 has gamma emissions that are not seen), while the A=236 derives from Pu-236 or U-236 (Np-236 has gamma emissions that are not seen). Neither U-232 nor U-236 is naturally occurring, therefore suggesting the possibility that the material has been irradiated in a reactor. Of course, the mass-232 peak could be due to a mixture of a small amount of U-232 with the remainder Th-232, which would suggest that the sample had been irradiated in a reactor and then recovered in a reprocessing plant and re-enriched. It should be pointed out that the majority of the small mass-232 peak could have originated from Th-232 despite not having detected Ac-228 in the sample, since the half-life of Th-232 is extremely long (14 billion years).

**Table 3.** Elemental and isotopic composition of HEU sample

LABORATORY:	DRDC OTTAWA	HEALTH CANADA		CNSC
<i>Analysis Method:</i>	<i>Gamma Spec</i>	<i>Gamma Spec</i>	<i>ICPMS</i>	<i>Gamma Spec</i>
<sup>234</sup> U (% by isotope mass)	0.84 ± 0.02	0.93 ± 0.04	1.02 ± 0.01	0.78
<sup>235</sup> U (% by isotope mass)	90.61 ± 0.72	90.1 ± 2.4	90.20 ± 0.04	85.79
<sup>238</sup> U (% by isotope mass)	8.55 ± 0.70	8.96 ± 0.80	8.78 ± 0.03	13.44
Total U (% by total mass)	75.1 ± 6.9	92.3 ± 2.3	79 ± 8	-
<sup>212</sup> Pb (Bq/g)	328 ± 33	472 ± 14	-	-
<sup>212</sup> Bi (Bq/g)	342 ± 13	491 ± 37	-	-
<sup>208</sup> Tl (Bq/g)	136.1 ± 3.4	152.8 ± 9.2	-	-

While the reports require that only definitive findings be included, a space is provided for discussion of speculative results. The above musings were included there.

The analysis results contained in the one-week report were adequate for identifying the seized material. The one-week report concluded that the material was weapons-usable highly enriched uranium. The controlled nature of this material would certainly provide investigators just cause in prosecuting the individual. Delays in shipment of the sample to three of the participating labs affected the quantity of results included in the report.

### 3.3 Two-month Results

The third and final report to the ITWG provided all information acquired in the forensic and laboratory analyses. Most of the laboratory work was complete within the first month of the start of the exercise, and the last month was spent interpreting the data and preparing the final report.

The forensic examination of the non-nuclear material seized as evidence in the first 24 hours of the exercise included fingerprint, DNA and trace element analysis. While a fingerprint was located on the seized vial, fingerprint processing turned up nothing of value. During the fingerprint processing, several of the fingerprint chemicals reacted with substances on the coaster (beer cardboard), suggesting the presence of biological material (i.e. DNA evidence). DNA examination of the evidence was negative for any DNA profiles. Furthermore, recovery of any usable trace evidence from the exhibits came up negative.

Physical characterization of the sample was not expanded beyond what was already included in previous reports. Both DRDC Atlantic and U of A performed ICMPS analysis consistent with the isotopic composition results of the other groups. DRDC Atlantic also performed a trace element analysis using ICPMS. Health Canada re-analysed their HPGc data to obtain an estimate of  $^{236}\text{U}$  content. The average of all such analyses, as given in the final report, are listed in Table 4. Unfortunately, no interpretation of the neutron activation analysis was made available by RMC.

As mentioned above, interpretation of the data was performed in an attempt to determine the origin of the material. Personnel in the Physics and Fuel Division of CNSC performed the interpretation. The data indicate that the material is "weapons-usable" HEU, enriched to 90%. Based on the presence of  $^{236}\text{U}$  and the lack of fission signature elements (Pu, Am, Cm), this material was most likely irradiated (producing  $^{236}\text{U}$ ) then reprocessed (removing activation products) and re-enriched for future use as fuel for a research reactor.

As far as the origin of the HEU, this fuel, most likely, comes from re-processed, re-enriched spent reactor fuel. The reason for this is the identified presence of a much larger quantity of U-234 than is found in enriched fuel derived from natural sources, and the presence of U-236, which does not exist in nature. In fact, U-236 is generated via (n,gamma) capture from U-235. Furthermore, U-234 is obtained via (n,gamma) capture from U-233, which in turn is created through the process of Th-232 (n,gamma)



**Table 4.** Composition of HEU sample.

	ISOTOPE	MEASURED VALUES
Uranium Concentration (% isotope mass)	<sup>234</sup> U	0.89 ± 0.09 %
	<sup>235</sup> U	90.2 ± 1.1 %
	<sup>236</sup> U	0.39 ± 0.38 %
	<sup>238</sup> U	8.54 ± 0.36 %
(% total mass)	Total U	81.3 ± 6.5 %
Other Isotopes (activity/ sample mass)	<sup>212</sup> Pb	400 ± 18 Bq/g
	<sup>212</sup> Bi	417 ± 20 Bq/g
	<sup>208</sup> Tl	144.5 ± 4.9 Bq/g
Trace Elements (% total mass)	Mg	0.001%
	Al	0.002%
	Fe	0.004%
	Cu	0.001%
	Zn	0.002%
	Sn	0.02%
	Te	0.005%

capture to Th-233, which then decays rapidly (half-life of 22.3 min) to Pa-233, which also decays rapidly (27 days) to U-233. So, the original spent reactor fuel must have contained Th-232 in addition to U-235. Such fuel is typical for HTGR reactors (i.e., High Temperature Gas Cooled Reactors).

Following use in an HTGR, the spent fuel was likely reprocessed to separate out the fission products from the uranium, resulting in uranyl nitrate. The uranyl nitrate would then be converted into UF<sub>4</sub>, which is then converted to UF<sub>6</sub>. The UF<sub>6</sub> would have then undergone a re-enrichment process to produce 90% U-235, and then been converted to UO<sub>2</sub>, with the ultimate goal of producing fuel for a research reactor.

Identification of countries that have HTGR reactors (such as UK and Germany) would narrow down the initial origin of the material. By identifying countries with reprocessing and re-enrichment facilities capable of producing such highly enriched material, and cross-referencing them with countries having either a HTGR facility or a research reactor that uses 90% enriched HEU as fuel, material origin could be determined. This process, however, was not performed for the exercise.

The final report was able to provide a more comprehensive identification of the seized material, in terms of elemental, isotopic, and trace element concentrations. The data interpretation suggested a possible original and current intended use of the material. However, forensic analysis of the non-nuclear material resulted in no usable information and the material origin was not ultimately identified.

## 4. Comparison to ITWG results

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As mentioned earlier, nine other countries performed the HEU round robin exercise from 2000 to mid-2002. The experience in response to real nuclear forensic investigations varied significantly from laboratory to laboratory, from no experience to upwards of thirty nuclear smuggling cases. The participating laboratories (or, in some cases, the lead laboratory for a given country) are listed below.

- ARC Seibersdorf GmbH, Seibersdorf, Austria
- AWE, Aldermaston, United Kingdom
- Commissariat à l'Énergie Atomique, Valduc, France
- Çekmece Nuclear Research and Training Center, Istanbul, Turkey
- Institute for Transuranium Elements, Karlsruhe, European Commission laboratory
- Institut für Radiochemie, München, Germany
- Institute of Nuclear Chemistry and Technology, Warsaw, Poland
- Institute of Isotope and Surface Chemistry, Hungary
- Lawrence Livermore National Laboratory, USA
- Lithuania Institute of Physics, Vilnius, Lithuania
- Nuclear Research Institute, Řež, Czech Republic
- Defence Research and Development Canada – Ottawa, Canada

Each of these laboratories was assigned an island name to allow for confidentiality of data and anonymity, in line with the fact that this exercise was meant as a learning experience not a performance evaluation. The code names were then used throughout the tests and in all correspondence concerning results and their evaluation. The remainder of this section will focus on comparing Canada's results with those of the rest of the ITWG. A detailed comparison of each of the three submitted reports on a question-by-question basis is included in Annex B. It should be pointed out again that the origin of the HEU material was undeclared and unknown to all ITWG exercise planners and participants, making a definitive assessment of each country's answers impossible.

Within the first 24-hour reporting period, the majority of laboratories were able to address questions about health and safety concerns, took precautions to preserve and collect fingerprint evidence, noted that the material in question was Highly Enriched Uranium (HEU), and some laboratories were able to obtain an initial indication of the



relative abundances of other uranium isotopes. All of the laboratories noted that the material in question constituted a hazard. The main oversight by labs was in the identification of some of the planted non-nuclear forensic evidence, such as the seeds and organic compounds present. Canada's response addressed all of these items, with the exception of the missed seeds, and thus was comparable to the other laboratories.

Within the first week, most laboratories were able to locate fingerprints, but the fingerprints were deemed unsuitable and insufficient for identification. A very accurate determination of the (final) isotopic and elemental analysis of the sample material was performed and the material was characterized as weapons-useable by all of the labs. Furthermore, potential applications and end uses were described for HEU of the quality and characteristics seen in the sample. For the one-week report, Canada was not able to provide any details on the non-nuclear forensic analysis due to delays at the RCMP forensic laboratory (because of a real investigation). Furthermore, two of the three ICP-MS analyses performed were not reported in the first week, thus the isotopic and elemental analysis given was not the final version. However, it is fully expected that in a real investigation this information would be available within the first week. In regards to the isotopic and elemental analyses, half of the laboratories (including Canada) did not perform plutonium isotopes on the sample. This is unfortunate, since the contamination identified on the radioactive vial's exterior turned out to be inadvertent plutonium contamination originating from the hot cell/glove box where the ten samples were prepared for this exercise. A comparison of Pu levels inside and outside the vial might have provided more clues about the sample's recent history.

Within the 2-months reporting period, initial measurements were confirmed and efforts were made to provide attribution of the sample. The laboratories were not able to identify the fingerprints introduced in the HEU Round-Robin Exercise and the laboratories largely did not detect the seeds/plant material evidence (Canada was no exception). Furthermore, multiple nuclear processes were attributed to the HEU.

The uranium isotopic abundances as reported by the laboratories are displayed in Table 5 (Canada's code name is Skye). It is interesting to note that the TIMS analytical results associated with the U-235 analysis were within 0.1% of each other. It was also noted that the multi-collector ICP-MS results were right in the midst of the TIMS values. Canada's U-236 value is low due to the values quoted by Health Canada, although the error value accounts for the spread. This points to a need for an intercomparison of Canada's analysis capabilities to prevent conflicting results. The format for presenting the analytical results (indication or no indication of the error range) was variable for the different laboratories.

Table 6 provides a summary of the nuclear processes suggested by each country. Many of the laboratories did not attempt to provide a country of origin because they had insufficient knowledge and/or databases to compare their nuclear forensic findings. Four laboratories reported on age dating of the uranium (date since last purification or processing of the material), which was determined to be February – July 1979. The uncertainty expressed by the four laboratories age dates overlapped indicating very good agreement.

**Table 5. HEU isotopic analysis results comparison (in atom %)**

LABORATORY	U-234	U-235	U-236	U-238	METHOD
Azores	0.97	89.99	0.68	8.37	LEGS, HRGS, ICP-MS, TIMS
Barbados	85.6 +/- 3.8 U-235				HPGe
Borneo	0.85 +/- 0.15	86.7 +/- 1.5	0.57 +/- 0.08	11.9 +/- 0.9	ICP-MS
Chatham	0.960 +/- 0.001	89.94 +/- 0.06	0.643 +/- 0.003	8.462 +/- 0.006	TIMS
Galapagos	0.96	89.89	0.68	8.47	TIMS
Mindanao	0.96 +/- 0.40	89.91 +/- 0.11	0.678 +/- 0.23	8.443 +/- 1.29	TIMS
<b>Skye</b>	<b>0.89 ± 0.09</b>	<b>90.2 ± 1.1</b>	<b>0.39 ± 0.38</b>	<b>8.54 ± 0.36</b>	<b>HRGS, ICP-MS</b>
Tobago	1.05 +/- 0.07	89.37 +/- 1.8	0.69 +/- 0.05	8.88 +/- 0.2	ICP-MS
Tonga	0.967 +/- 0.001	89.99 +/- 0.02	0.679 +/- 0.001	8.362 +/- 0.005	TIMS
Trinidad	0.955 +/- 0.001	90.01 +/- 0.01	0.673 +/- 0.001	8.365 +/- 0.004	MC ICP-MS
<i>Mean (not including Skye)</i>	<i>0.96 +/- 0.16</i>	<i>89.0 +/- 1.4</i>	<i>0.66 +/- 0.09</i>	<i>8.91 +/- 0.56</i>	

**Table 6. Attribution of HEU material history - Nuclear processes**

LABORATORY	NUCLEAR PROCESS
Azores	Reprocessed fuel/ Enrichment plant
Barbados	Enrichment convert to U <sub>3</sub> O <sub>8</sub>
Borneo	Material test reactor, submarine reactor
Chatham	Special enrichment plant
Galapagos	Gas diffusion plant
Mindanao	Sol-gel precipitation
<b>Skye</b>	<b>Reprocessed, re-enriched spent reactor fuel</b>
Tobago	Secondary enrichment after reprocessing
Tonga	Reprocessed fuel
Trinidad	Chemically reprocessed U oxide

Discussion on the HEU Round-Robin Exercise by the ITWG highlighted several shortfalls, provided below [2].

From a procedural standpoint, of predominant concern was the trace contamination that several laboratories identified from the preparation of the samples for the exercise. It could be demonstrated that the contamination had inadvertently occurred during the preparation of the samples. Contamination



control is obviously of critical concern for the laboratories involved in this round-robin exercise and in real cases. Another feature of running a “legitimate” blind experiment is the transport and receipt of “unknown” samples to a laboratory when most shipping requirements entail disclosing what is being shipped. Finally, the specific material used in this exercise had unknown origin and pedigree so that feedback to the labs could not be reasonably and readily applied.

From a technical standpoint, the results from this exercise points out the importance and lack of a comprehensive network of data and knowledge bases to assist in the interpretation of the data in light of nuclear processes and country of origin. Also, from a classical forensic technologies standpoint, it was obvious that a more close relationship needs to be developed with the law enforcement community to integrate nuclear forensics into the overall scheme of application of forensic techniques and methods to a crime scene. Laboratories need to be generally sensitive to other “classical” forensic methods and to avoid the potential for contamination of evidence for other techniques. Also, efforts need to be made to develop, compile, and share databases and knowledge bases associated with characteristics of nuclear processes with various countries.

These comments are certainly in line with many of Canada’s observations in relation to the exercise.

Another area of discussion for the ITWG dealt with the prioritization of methods and techniques that provided the best results for a given reporting period. The results from the ITWG discussions are provided in Table 7 below.

**Table 7. Technique and method prioritization for HEU round robin exercise**

TECHNIQUES/METHODS	24-HOUR	1-WEEK	2-MONTH
Radiological	Dose Rate ( $\alpha, \gamma$ , neutron) Surface Contamination Radiography of vials		
Physical Characterization	Visual Inspection Photography Weight Dimension Optical Microscopy Density	SEM (EDX) XRD	TEM (EDX)
Classical Forensics	e.g., Fingerprints		
Isotope Analysis	$\gamma$ -Spectrometry $\alpha$ -Spectrometry	MS (SIMS, TIMS, MCMS)	
Elemental/Chemical		ICP-MS XRF IDMS	Ion Chromatography

## 5. Discussion

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Nuclear forensic analysis and nuclear attribution are increasingly important tools in addressing the illicit trafficking of nuclear and radiological material. In light of the increased threat of an incident of radiological terrorism occurring, development of procedures and protocols for responding to the interdiction of nuclear and radiological materials is of great importance. Participation in this international exercise brought together several Canadian nuclear analysis laboratories to test existing procedures for such a response. Inclusion of the RCMP in the round robin also exercised the link between law enforcement responders and the laboratories.

The RCMP's participation in this exercise was important since a real incident would most certainly require some amount of forensic investigation. While no useful evidence was ultimately identified, exercising the response to such an incident proved useful. In fact, the issue of dealing with radiologically contaminated forensic evidence was raised in the context of the exercise and protocols were set up as needed, proving the usefulness of the round robin. Follow-up for the exercise will include defining and implementing procedures for handling and processing such evidence.

DRDC Ottawa performed sample receipt, distribution, and analysis of the HEU. DRDC Ottawa also compiled the results from all of the participating labs and wrote the ITWG reports. Protocols for handling a powdered radioactive material were developed and tested, using a sealed glove box to contain any scattered material. Many of the nuclear analyses proposed for the sample proved ineffective either due to the nature of the sample or the lack of sufficient sensitivity of the proposed analysis. For example, neutron spectrometry was performed but the lack of a sufficient quantity of neutron emitters in the sample yielded a null result. Also, alpha spectrometry measurements were not able to identify any alpha-emitting isotopes due to the high degree of alpha absorption in the sample, producing significant energy dispersion.

The most useful analysis performed at DRDC Ottawa proved to be high-resolution gamma spectrometry with the high purity germanium detector. This analysis allowed for the determination of uranium isotopes, total uranium concentration, and activities of several other isotopes present in the sample. The data were consistent with those of the rest of the groups, as well as the final values reported by the ITWG. Following completion of the exercise, DRDC Ottawa developed procedures for performing age determination of the HEU sample by comparing the activity ratio of  $^{234}\text{U}$  to  $^{214}\text{Bi}$ . An age of  $27.0 \pm 1.7$  years was determined, yielding a date of last purification or processing of the HEU between November 1975 and March 1979. This coincides somewhat with age dating estimations of the rest of the international round robin participants, who reported dates between February and July 1979.

Health Canada received the sample on day 2 of the exercise and proceeded to perform physical characterisation via scanning electron microscopy (SEM) as well as isotopic and elemental characterisation via both HPGe gamma spectrometry and ICPMS analysis. Health Canada was by far the most comprehensive in their results reporting.



The ITWG reporting forms were submitted by HC to DRDC Ottawa at 24 hours, one week and one month following sample receipt at HC. While the majority of reported results were consistent with those of the other labs, two values quoted in the HC results were significantly different from the results of the rest of Canada and of the ITWG. The % total uranium concentration as determined by HPGe gamma spectrometry was more than 10% different from all other measurements. However, HC measured this value by two other methods (ICP-MS and kinetic phosphorescence), both of which yielded values consistent with the rest of the participants and not with its own HPGe measurement. Also, the  $^{236}\text{U}$  concentration as measured with ICP-MS and HPGe gamma spectrometry underestimated the value by more than 50% compared to ICP-MS values quoted by two other laboratories and the final values of the ITWG. While the gamma spectrometry data was the result of a deconvolution of two over-lapping peaks, which could explain the underestimation, ICP-MS values cannot be explained at this time.

The CNSC laboratory also received the sample on day 2 of the exercise. A single report was submitted to DRDC Ottawa during the first week following receipt at the laboratory. This report described both the gross gamma activity with a well gamma counter and  $^{235}\text{U}$  determination via HPGe gamma spectrometry. Other isotopes that were identified by gamma spectrometry were also listed. Unfortunately, little of this information was of use in the characterisation and attribution of the HEU sample. Since the completion of the exercise, a more detailed HPGe gamma spectroscopy analysis was performed and the isotopic composition from this analysis is consistent with the other groups. The CNSC laboratory should have performed this level of analysis during the exercise.

RMC received the sample on day four of the exercise following delivery by a courier. The sample was delivered one day later than expected, and when the driver arrived, RMC personnel (wearing a white lab coat) met the driver outside. The driver failed to ask for ID or to check that the person to whom he handed the package was indeed the intended recipient. This delivery highlighted an interesting aspect with regards to safety and security of radioactive materials, as well as the need for an escort in a real situation.

Apart from the delivery incident, RMC submitted one report to DRDC Ottawa within the first week following receipt at the lab. This report indicated that gamma spectrometry, liquid scintillation counting, and neutron activation analysis had been performed. Although no quantification was performed, the gamma spectrometry analysis identified the sample as predominantly  $^{235}\text{U}$ . LSC analysis also did not provide the necessary information required for sample characterisation. While neutron activation analysis (naa) was performed and the first (and only) RMC report stated that the presence of fissile content was identified, no interpretation of the naa data was ever submitted. Ultimately RMC was not able to provide any unique information regarding the sample.

The University of Alberta SLOWPOKE Facility received the sample on day five of the exercise, also via a courier. This package arrived two days later than expected. Two reports were submitted by U of A, the first at 24 hours following sample receipt, and

the second at one month. Analysis data provided in these reports included HPGe gamma spectrometry analysis and ICP-MS, which were consistent with the other laboratories. A detailed description of the analysis procedures was also included in the report, making reporting of the results to the ITWG much easier.

Delivery of the sample aliquot to DRDC Atlantic – Esquimalt Division took the longest and suffered the greatest delays. The sample arrived on day six of the exercise via courier, three days later than expected. Two reports were submitted, the first at 24 hours following sample receipt, and the second at one week. Analysis data provided in these reports included ICP-MS and SEM, which were consistent with the other laboratories. Other analyses (XRD) were planned for the sample but were prevented by equipment problems. The ICP-MS results from Esquimalt were the only ones to include trace element concentrations.

An overview of the analyses proposed by each laboratory before commencement of the exercise is given in Table 8, along with the analyses actually performed and the usefulness of the analysis. The fact that these analyses were part of an exercise and not a real-case situation likely addresses many of the issues raised in this discussion. It cannot be expected, for instance, that personnel at seven different laboratories should drop everything to process the round robin material. That said, reporting of findings should be done in a more thorough manner, with procedures and accuracy included, so as to assist the person compiling all of the results for further reporting.

**Table 8.** *Analyses promised, performed and used in the exercise reports*

LABORATORY	PROPOSED ANALYSES	PERFORMED ANALYSES	COMMENTS
DRDC – Ottawa	Sample receipt and distribution; Radiological analysis; U isotopics (gamma spec)	Sample receipt and distribution; Radiological analysis; U isotopics (gamma spec)	Some radiological analyses not useful; U isotopics used
RCMP	Forensics evidence processing (fingerprints, DNA, trace materials)	Forensics evidence processing (fingerprints, DNA, trace materials)	No usable evidence found, but analysis useful
CNSC	U isotopics (gamma spec); Interpretation	U isotopics (gamma spec) Interpretation	U isotopics not fully analysed; interpretation useful
Health Canada (HC-RPB)	U isotopics & trace elements (gamma spec, ICPMS); Particle composition and morphology	U isotopics (gamma spec, ICPMS); Particle composition and morphology	Trace element analysis not performed; U isotopics had some analysis discrepancies
RMC SLOWPOKE Facility	Neutron activation analysis	Neutron activation analysis	No interpretation performed so no results given
UofA SLOWPOKE Facility	Neutron activation analysis; U isotopics (gamma spec, ICPMS)	U isotopics (ICPMS)	U isotopics were used
DRDC – Atlantic Esquimalt Division	U isotopics & trace elements (ICPMS); Particle composition and morphology	U isotopics & trace elements (ICPMS); Particle composition and morphology	All analysis results used



## 6. Recommendations and Conclusions

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Performing the initial forensic assessment on the material at the receiving laboratory proved to be quite effective for this scenario. The commingled radiological and forensic evidence required that the initial package opening include an assessment for radiological contamination on all material prior to it being taken for non-nuclear forensic analysis at the RCMP laboratory. In an ideal situation, all forensic and nuclear analyses would be processed at the same lab. However, none of the participating Canadian labs have such facilities and thus performing the initial investigation at a location where the radiological assessment can be performed is vital. Unfortunately, some potentially critical non-nuclear forensic clues were missed in this analysis (i.e. the presence of the pollen seeds), which may have been detected if the vial had been processed at the forensics laboratory. Protocols for handling radiologically contaminated forensic evidence also need to be worked out and codified.

Minimisation of the nuclear analysis laboratories involved would simplify both the shipping requirements and results reporting. This exercise helped in identifying the laboratories with the greatest range of analysis capabilities. DRDC Ottawa and Health Canada could most effectively perform future forensic analysis of an unknown seized material, with the ability to contract RMC to perform neutron activation analysis if necessary. This localises the analysis to a small area, minimizing transport requirements, allows a large number of analyses to be covered while facilitating data compilation and reporting of results. An alternative combination of DRDC Atlantic – Esquimalt Division and the University of Alberta SLOWPOKE Facility would provide a similarly adequate capability in the west of Canada, however, sample processing and receipt has not been exercised at either of these locations. Participation by CNSC would be mandatory to ensure that all licencing requirements are met and to perform data interpretation.

To ensure that the analysis results are consistent across all participating laboratories, inter-comparisons should be performed on a regular basis. This could be via Canadian, US/CA, or international round robins. In the meanwhile, Health Canada should investigate the discrepancies seen in their round robin analysis results to determine the origin. A further aim of future inter-comparisons is to solidify the links between different groups (law enforcement, laboratories, etc.). Inclusion of other groups, such as customs/border agents or legal counsel, might also prove beneficial. Identification of analysis laboratories with further capabilities for participation in these future exercises would also strengthen Canada's nuclear forensic response. Reporting requirements should be defined for any future exercise and every participating lab should follow these recommendations in order to facilitate data compilation for overall reporting.

In a real nuclear forensic incident, only licenced and authorized carriers should be used to transport the material [3]. If possible, however, either law enforcement officials or nuclear regulators should transfer the illicit material. This would ensure delivery in a timely manner to the intended recipient. Furthermore, if scientific evidence resulting

from the analyses requires presentation in a court of law, chain of custody of the material must be thoroughly tracked to ensure safety, security and preservation of evidence. Keeping said material in the hands of regulators or law enforcement would address this requirement.

## 7. References

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1. NUCLEAR SMUGGLING INTERNATIONAL TECHNICAL WORKING GROUP (ITWG), Terms of Reference, June 1997. (Internal ITWG paper available on request).
2. Dudder G, Hanlen R, Herbillon G. "International interlaboratory forensics exercise with a highly enriched uranium sample (HEU round robin exercise) final report." Pacific Northwest National Laboratory Report PNNL-14698, June 2004.
3. Kristo MJ, Smith DK, Niemeyer S, Dudder GB. "Model action plan for nuclear forensics and nuclear attribution." Lawrence Livermore National Laboratory Report UCRL-TR-202675, March 2004.



## Annex A - Scenario

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A detailed scenario was incorporated into this exercise in order to introduce elements of a real nuclear smuggling case. The scenario, outlined below, included people, places, and organizations that were consistent with the presence of common forensic elements (the fingerprints, seeds, plastic shopping bag, and drawing on a beer coaster) that accompanied the HEU sample. The scenario provided to exercise participants was as follows:

On Tuesday 14 November 2000 around 11 a.m., a car was stopped at a petrol station located on the motorway precisely at the border between Luxembourg and Germany. The (male) driver was alone; he was in possession of a Belgian identity card and a driver's license, which, in the meantime, have proven to be false. Amongst other details, these documents mentioned the following identity:

Name : Luc, Marcel, Robert REMOISY  
Street : Rue de la Régence, 1, boîte 3  
City : 4000 Liège  
Born : 8 April 1956 in Brussels.

He did not have any other documents (such as a credit card) with him at the time he was arrested. He was holding some 14,500 Belgian francs and 2,000DM cash.

Whilst the name of the man is now proven to be false, the address does exist but corresponds to an old 7-floors apartment building abandoned since more than a year and that will be destroyed in order to build a shopping mall. His mother tongue is unknown but he is fluent in French with a Slavonic accent; he also seems to understand German and English. His real identity is still not known at this moment but his fingerprints can be found in the attachment (see Figure 6).

The man attracted attention because on 13 November he had contacted with his cellular telephone a small company located in Luxembourg and called « Nucleon Trade International (NTI) ». He said - in French - he could offer very high quality nuclear material suitable for atomic explosive devices at a reasonable price and within very short delay. The man spoke about "high quality" uranium and of some 20 kilos. He also said that he could provide a sample to give evidence of what he was offering.

Note: The reason why the man contacted this company in particular is still unclear. This company exists since 1975 and is specialised in import/export, in particular with the middle and far-east. Before 1991 the company had also some lucrative business with the former Soviet-Union in high-tech equipment and components suitable for use in civil nuclear reactors. It appears that the company had abandoned this kind of business since at least 5 years.

--- THIS IS AN EXERCISE --- THIS IS AN EXERCISE ---

ITWG ROUND ROBIN TEST

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**FICHE de relevé des empreintes digitales**


Date: 24/11/2000

Référence: Offre suspecte d'assassinat


Nom du prévenu: L. REMOISY (faux ?)

Sexe: H

Age: 45 (?)




Pouce gauche



Pouce droit

Nom de l'agent et signature: A. R. GONAUT



**Figure 6.** Fingerprints of suspect taken on the scene.

When receiving the phone call from the so-called Mr Remoisy the manager of NTI convened an appointment at the premises of the company at 3 p.m. on Monday 13 November. Moreover, the manager also informed the local police who took the necessary measures in order to intervene eventually and to interrogate the man. However the man did not show up.

Before concluding that this was a hoax, the police made an investigation in the hotels in and around Luxembourg City. The trace of a certain Mr Remoisy could be found in a middle class hotel near a large supermarket. Appropriate measures were taken to keep him under surveillance.

When he was stopped on the motorway the next day (November 14th) and invited to park his car on the nearby parking spot the man became very nervous. Besides half a dozen of bottles of strong alcohol and 8 packs of Camel cigarettes bought in the supermarket nothing appeared unusual at first glance. However during the body search a creased plastic bag was found in one of the pockets of his jacket. This bag contained 2 small vials suitable to contain nuclear material or/and other radioactive substances. The bag also contained a "beer cardboard" with a little self- handmade drawing on one side (see Figure 7). Besides his wallet and this plastic bag, the police also found a cellular telephone in his pockets. The phone was operating with a prepaid phone card.





**Figure 7.** Pictures taken on the scene. (a) Confiscated material. (b) Inside view.

The police decided to call a local expert on the scene. The radiation protection specialist arrived on site within the hour with appropriate equipment. He confirmed on the scene that the “plastic bag” was emitting gamma radiation. A significant peak in the 186-keV region seemed to indicate a predominant presence of uranium-235. However the dose rate outside was not so high that it could bring the carrier’s health in danger. There was also no evidence of any contamination on the outer part of the bag.

The man was then immediately arrested and the car and all his belongings seized. When questioned on site on the reason for carrying this sort of objects, on the purpose of it, on the reason of his presence in Luxembourg and on why he was going to Germany the man remained silent.

Later in the day the phone calls he had given since he had been on the Luxembourg territory were identified: no call from the hotel with the room telephone, 3 from his cellular phone, all to the same number in Germany to another cellular telephone working on the anonymous prepaid card principle. When the police called this German number the first time, a man saying a few words in a Slavonic language replied but then rang off. Later trials were unsuccessful. The user of that telephone could be localised as receiving the calls in the Frankfurt-city area. Obviously this was not sufficient to find and arrest this presumed accomplice.

On recommendation of the local expert and considering the low level of radiation, the plastic bag was properly wrapped in plastic and put into a type A transport container. It was immediately routed to the nearest located installation able to cope with radioactive substances. There, more precise non-destructive assays were performed and they confirmed the presence of high-enriched uranium.



This was the state of affairs on the day of seizure at 6 p.m. The police and other competent authorities then decided to request extensive and forensic analysis. The necessary contacts were taken and appropriate authorisations given. The “plastic bag” and its content were carefully repacked and prepared ... for transfer to your laboratory with a formal request for extensive forensic analysis!

Note: While some dates were used in this simulated scenario, they are of course irrelevant for the purpose of the analytical part of the exercise. Therefore, please imagine that you have actually received the sample in your laboratory on the day following the seizure.

## Annex B – ITWG Expectations

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The following was compiled by the ITWG Exercise Task Group and has been amended to include Canada's response.

### Questions and Expectations for the 24-Hour HEU Round Robin Report

ITWG recommended that the following methods be given priority during the first 24 hours after receipt of the sample in the laboratory.

- Physical Characterization: visual inspection, weight, dimensions, photography, optical microscopy
- Radiological: contamination, total dose, dose rate (gamma, alpha, and neutrons)
- Elemental and isotopic analysis: gamma and alpha spectroscopy

#### Overall organizer expectation for first 24 hours:

Focus: Safety and determination of material (hazards, legal issue, and immediate threat)

#### Organizer's Expectation:

- Determine that the material is HEU
- Ensure that other potential non-nuclear forensic evidence are protected
- Ensure that health and safety procedures are in place and considered

#### Lab capability expressed (respectively):

- HEU – 8 of 9 yes, 1 presence – Canada: Yes
- 7 of 9 yes, 1 partial, 1 no – Canada: Yes
- 6 of 9 yes, 3 partial – Canada: Yes

### Questions

1. By which means did the "parcel" arrive? Escorted by whom?

Organizer expectation: There would be a procedure to contact officials or authorities.

Lab capability expressed: 8 of 9 yes – 1 lab with detailed protocol, 1 no apparent escort – Canada: Yes

2. What did happen when the seized "parcel" arrived at your laboratory (before opening)?

Organizer expectation: There would be procedures for safety, preservation of non-nuclear evidence, including a considerations for booby-traps, containment and contamination control.

Lab capability expressed: 6 of 9 yes, 1 partial, 2 delayed – Canada: Yes

3. Give a detailed description of the parcel and the opening procedure. What did the content look like?  
Organizer expectation: There would be a procedure for photographing and describing the physical evidence of the materials provided and procedure for maintaining the integrity of the evidence  
Lab capability expressed: 9 of 9 yes – Canada: Yes
4. Have specific measures taken to look for fingerprints or other important elements when opening the parcel?  
Organizer expectation: A procedure or description of the specific measures applied to look for non-nuclear evidence.  
Lab capability expressed: 6 of 9 yes, 1 simulation, 1 delayed, 1 no – lab only wished to assay nuclear material – Canada: Yes
5. What is the material? (physical characteristics, major elemental and isotopic characteristics)  
Organizer expectation: A physical description (color, size, powder, etc.) of the materials, presence of HEU, and presence of seeds, pollen, or other material.  
Lab capability expressed: 9 of 9 yes – applies to nuclear materials, 3 of 9 noted presence of seeds – Canada: Yes; however seeds, pollen, etc. were not identified
6. Other material characteristics? (Unique characteristics)  
Organizer expectation: Any additional observations  
Lab capability expressed: 8 of 9 yes, 1 no response – Canada: Yes
7. Method(s) used?  
Organizer expectation: Documentation of the method used. Were the ITWG suggestions (above) were followed?  
Lab capability expressed: 9 of 9 generally yes, when able to do so. – Canada: Yes
8. Does this material present a hazard or a threat (health safety, and potential applications)?  
Organizer expectation: An assessment of the health and safety considerations for the material is provided.  
Lab capability expressed: 9 of 9 yes – Canada: Yes
9. Other findings?  
Organizer expectation: Expected to describe the vials, “yellow” dot, etc. and note that organic compounds may be present in the “Beer Coaster”.  
Lab capability expressed: 2 of 9 yes to the dot (4 labs eventually), 1 of 9 noted organic compound, 5 no response, 4 labs describe vials further – Canada:



Provided an analysis of 50 year committed effective doses. Neither yellow dot nor organic compounds were mentioned.

10. (Non-forensic question) Is handling, storing, or selling of this material governed by legal provisions in your country? (technical opinion)

Organizer expectation: Statement that “yes” it is covered.

Lab capability expressed: 9 of 9 yes – Canada: Yes

### Questions and Expectations for the 1-Week HEU Round Robin Report

ITWG recommended that the following methods be given priority during the first week after receipt of the sample in the laboratory.

- Physical characterization: additional visual inspection, weight, dimensions, photography, optical microscopy, and SEM (scanning emission microscopy)
- Isotopic Analysis: TIMS (thermal ionization mass spectrometry), alpha and gamma-spectroscopy
- Elemental and chemical analysis: for instance ICP-MS (inductively coupled plasma-mass spectrometry)

Overall organizer expectation for first week:

Focus: Confirmation of 24-hour results and development of other leads

Organizer’s Expectation:

- Confirm that the material is HEU with Full U isotopics.
- Identify other nuclear indicators (Pu isotopics, other elements present)
- Identify other non-nuclear evidence and obtain information from other forensic evidence recognized in first 24 hours

Lab capability expressed (respectively):

- 9 of 9 yes – Canada: Yes
- 8 of 9 yes, 1 no – Canada: Yes, but not Pu isotopics
- 1 of 9 yes, 1 partial, 7 no – Canada: No

### Questions

1. Give a detailed description of the parcel and the opening procedure if this operation did not take place during the first 24 hours. What did the content look like?

Organizer expectation: If not expressed in 24-hour report, there would be an indication of a procedure for safety, preservation of non-nuclear evidence, considerations for booby-traps, containment and contamination control. There would be a procedure for photographing and describing the physical evidence of the materials provided.

Lab capability expressed: All labs reporting Yes. – Canada: Yes

2. Have specific measures been taken during the week to look for fingerprints or other elements relevant from a forensic point of view (hair, pollen, dust, etc.)? Please report the results if any.  
Organizer expectation: Expected to find fingerprints and match them up, assess pollen/seeds, assess the organic compounds present in the “beer coaster”, precautions taken to preserve evidence.  
Lab capability expressed: 1 no response. 2 yes, 2 fingerprints not suitable, 3 not yet done or delayed – Canada: Yes, but analyses not complete
  
3. What is the material? (physical/chemical characteristics, mixture or not, major and minor elemental and isotopic compositions, unique manufacturing characteristics, “age” of the product)  
Organizer expectation: A detailed physical description (color, size, , powder, , etc.) of the materials, Assay of HEU, confirmation of other elements present in sample, isotopic composition (abundance) of isotopes for actinides found.  
Lab capability expressed: 9 of 9 labs yes – Canada: Yes
  
4. Other material characteristics? (unique characteristics)  
Organizer expectation: Any further details of the physical form of the material.  
Lab capability expressed: 5 of 9 yes, 4 no response – Canada: Yes
  
5. Method(s) and information references used? (description, precision, sequence used, particular features)  
Organizer expectation: Sequenced order of analysis stated, and citation of database used for comparison,  
Lab capability expressed: 7 of 9 yes, 2 not clear; data base not clear – Canada: Yes
  
6. Can the material be classified as weapons grade material?  
Organizer expectation: Recognize that the material is “weapons-useable” material.  
Lab capability expressed: 9 of 9 yes – Canada: Yes
  
7. What is the material used for or what can the material be used for? (legitimate end-use, potential applications)  
Organizer expectation: State potential applications for the material.  
Lab capability expressed: 9 of 9 responded – Canada: Responded
  
8. Other findings? (refers also to additional information you want to give concerning hazard and threat)  
Organizer expectation: Identify additional efforts made to describe unique characteristics and the hazard or threat.



Lab capability expressed: 5 of 9 responded, 4 no response – Canada:  
Responded

9. At this stage, please indicate how you intend to continue your work to get comprehensive forensic work? (techniques you want or would wish to use)  
Organizer expectation: A plan or procedure of what additional work can be done (techniques and analysis applied) over the next seven weeks to confirm and provide unique features of the nuclear and non-nuclear evidence for source attribution.

Lab capability expressed: 9 of 9 respond yes – Canada: Yes

### **Questions and Expectations for the 2-Month HEU Round Robin Report**

The purpose of this “final” report is to provide comprehensive forensic analysis of the materials and accompanying items to provide leads for investigation and evidence for prosecution. This means, inter-alia, that this report must concentrate on

- Developing the analytic methods and results
- Interpreting the results in the context of available information
- Prioritizing forensic methods and techniques
- Examining the utility of existing knowledge and/or databases.

Overall organizer expectation at 2 months:

Focus: Attribution.

Organizer’s Expectation:

- Identify the unique features and potential sources of HEU
- Confirmation and analysis of other nuclear indicators found in week 1.
- Confirm, analyze and report on information from other non-nuclear forensic evidence.

Lab capability expressed:

- 9 of 9 yes – Canada: Yes
- 7 of 9 yes, 2 no – Canada: Yes
- 2 of 9 yes, 7 no – Canada: Yes

### **Questions**

1. Have specific measures been taken after the first week to look for fingerprints or other elements relevant from a forensic point of view (hair, pollen, dust, etc.)? Please report the results if any.

Organizer expectation: A report on the findings of the fingerprints and pollen/seeds assessment.

Lab capability expressed: 6 of 9 fingerprints not found or not suitable for analysis, 1 simulated, 1 delayed, 1 only intended to work with nuclear



**forensics** – Canada: Fingerprints & organic compounds found but not suitable for further analysis

2. What is the material? (physical/ chemical characteristics, mixture or not, major and minor elemental and isotopic compositions, unique manufacturing characteristics, morphology, trace analysis, production process and process history, fingerprinting, evidence of origin, etc.)

Organizer expectation: Provide definitive statement of the material – the nuclear material composition, the seeds, the fingerprints

Lab capability expressed: 9 of 9 on nuclear forensics – Canada: Yes on nuclear forensics

3. What is the material used for, or could be used for? (legitimate end-use, potential applications, weapons useable material)

Organizer expectation: Concise statement of the most likely uses of the nuclear materials

Lab capability expressed: 9 of 9 responses – Canada: Responded

4. What was the source of the material? (Specific origin, production process, material production facility, nuclear fuel cycle, supply of more material)

Organizer expectation: Concise statement of the most likely source of the material

Lab capability expressed: 9 of 9 responses – Canada: Responded

5. Who was involved in the illicit trafficking? (thieves, sellers, brokers, buyers, organizations)

Organizer expectation: Recognition that this cannot be determined from the data without a comparison of fingerprints and other non-nuclear evidence by Interpol and other competent authorities.

Lab capability expressed: 6 of 9 repeat story or no response, 2 say thieves, 1 says individual – Canada: Repeated story

6. What was the illicit route? (Where has the material been)

Organizer expectation: Recognition that the non-nuclear evidence lends itself to this aspect. (Where did the seeds come from?)

Lab capability expressed: 8 of 9 repeat story or no response, 1 claims additional forensic evidence implicating EU laboratory – Canada: Repeated story

7. Other comments or findings?

Organizer expectation: Further description of laboratory efforts or statements about the material in question

Lab capability expressed: 4 of 9 no response, 5 some response – Canada: No response

8. Method(s) and information references used? (description, precision, sequence used, particular features)

Organizer expectation: Complete listing of techniques, generic instruments, databases, etc., used to arrive at conclusions from evidence.

Lab capability expressed: 9 of 9 responded – Canada: Responded

9. Please indicate here which techniques you would like or have liked to use to continue your work to get comprehensive forensic work?

Organizer expectation: What analyses would they like to have completed, but did not have the time, resources, or instruments to complete. Statement from labs regarding their sense of their own technology and analysis related gaps for conducting this sort of assessment.

Lab capability expressed: 7 of 9 responses, 2 no response. – Canada: Responded

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In the framework of combating the illicit trafficking of nuclear materials and other radioactive substances, the ITWG organised an international inter-laboratory round robin dealing with a thwarted illicit trafficking incident of a high-grade sample of highly enriched uranium (HEU). DRDC Ottawa coordinated Canada's response to the exercise, which included the participation of seven organisations, including this lab. The exercise involved a thorough forensic examination and analysis by the Royal Canadian Mounted Police, nuclear characterisation of the material by six laboratories, and interpretation of the nuclear data for attribution of the materials origin by the Canadian Nuclear Safety Commission. Overall, the analysis was successful in identifying the material and providing a reasonable interpretation of the data. This report outlines Canada's response, the lessons learned, and the way ahead in terms of combating illicit trafficking and nuclear smuggling in Canada. A comparison of Canada's results to those of the other participating countries is also presented.

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Nuclear smuggling, nuclear forensics, laboratory analysis, forensic analysis